OCT 1 2 2001   10/12/01

VALIDATION RESULTS FOR METHOD NO. Covance Harrogate 2001-032-D1

		MEAN	RELATIVE STD. DEV.(%)	RANGE	SEE (conc)	
ANALYTE	MDA					
MATRIX	Human plasma					
DATE	15-Aug-01					
LLOQ	(Provide acceptance Criterion)	0.4 µmol/L. Accuracy 107.5%. RSD 6.0%				
STANDARD ERROR OF ESTIMATE OF QC SAMPLES (concentration units) (SEE = S.D./SQRT (# of determinations))						
	1 µmol/L	Low QC			0.012	
	4 µmol/L	Med QC			0.034	
	8 µmol/L	High QC			0.068	
DESCRIPTIVE INFORMATION ON INSTRUMENT RESPONSE						
Function (Linear, Quadratic, etc.)	Linear					
Slope (mean)	67927					
Intercept (mean)	7935					
Other (mean regression value)	0.98855(1/x weighted)					
RECOVERY (as a %)						
High QC in (matrix)=	8 µmol/L	92.5%	Calculated as a % of the peak area observed for derivatised water standards			
Med QC in (matrix)=	4 µmol/L	94.7%				
Low QC in (matrix)=	1 µmol/L	100.0%				
Results with different sample amounts (results of QC evaluations) intra-assay						
LLOQ QC=	0.4 µmol/L	0.43	6.0%	0.39-0.45	0.013	
Low QC=	1 µmol/L	0.94	4.1%	0.90-1.00	0.016	
Med QC=	4 µmol/L	3.90	2.2%	3.79-4.05	0.035	
High QC=	8 µmol/L	7.55	2.4%	7.34-7.82	0.075	
Results with different sample amounts (results of QC evaluations) inter-assay						
LLOQ QC=	n/a	n/a	n/a	n/a	n/a	
Low QC=	1 µmol/L	0.95	6.1%	0.84-1.10	0.012	
Med QC=	4 µmol/L	3.84	4.3%	3.54-4.15	0.034	
High QC=	8 µmol/L	7.50	4.5%	6.85-8.10	0.068	
STABILITY (assuming any degradation is linear):						
a) Room Temperature in Matrix (+/- %/12 hrs)		7.7% maximum				
b) Freeze/thaw (+/-% /3 cycles)		3.3% maximum				
c) Processed sample (+/- %/24 hrs)	TBA	% maximum				

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Headings

TA	MPA	Study	44.000
Units	umol/l	Cal 1	0.4
Species	human	Cal 2	0.6
Matrix	plasma	Cal 3	1
Regula va	asc.	Cal 4	2
SPS	7	Cal 5	
OR/zo a begin ratio	1	Cal 6	5
LLOQ QC conc	3.4	Cal 7	7.5
Lo QC conc	1	Cal 8	9
Me QC conc	4	Cal 9	10
Hi QC conc	8		

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Footnotes from SOP 66 Rev 1: Batch annotation

   = Above data system
   = Anomalous result
BDS = Below data system
CO = Confirms original result
DNU = Data not used
DNR = Data not required
HI = High internal standard
M = Instrument malfunction (add reason if known)
P = Interferent peak
S = Insufficient sample
LI = Low internal standard
M = This result is a median value
ME = Mis-extraction
MI = Mis-injection
NR = No result
NS = No sample taken/received
OR = Over range
PC = Poor chromatography
PF = Poor fit with rest of regression (excluding standard improves the correlation coefficient / curve fit)
PF = Poor fit, calibration standard outside acceptance criteria
PP = Positive predose
PS = Poor sensitivity
SA = Sample dropped/spilt/spoilt during preparation (Lab accident)
SE Spiking error
UR Under range (when LLOQ reset)

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